

## 10.3.2 User Newsletter

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This is the fifth user newsletter for Beamline 10.3.2 and the first since the big April upgrade. Fortunately, I've good news to report.

### **Optics**

The new monochromator works very well. The flux is up by 3-10x (I haven't really measured it) and it's really stable. A big cheer is in order for the people who worked on it, Keith Franck, Tom Miller, Alastair Macdowell Greg Morrison, Bob Sublett, Ed Domning and Rich Celestre. If I left anyone out, I apologize in advance. Crystal tweak, roll and translation are done using a Cal-Bay MRAflex picomotor controller, which is an interface box between NI motion control boards and picomotors and LVDTs. One such box does 4 channels, so is enough for the mono. You hardly ever have to touch the tweak, even when going from very low energies up to the top, unless you want to detune for harmonic suppression. The one downside is that the resolution depends on the vertical roll-slit size and how well M2 is tuned. With small slits, the blip at the Cu edge shows as a blip, while it's a flat shoulder with full beam. Good enough for EXAFS and most XANES. The mono has been demonstrated to work all the way down to the P K edge.

The beam microscope has been built. It is now possible to see the focus directly, but at the cost of moving the sample stage. However, mirror tuning is still manual as we only bought one MRAflex.

We cleaned M2 and put it back in. Unfortunately, some Ga got on it from the cooling block and I was only partially successful in removing it. I think this contributes to a significant scatter which causes the beam to have a long vertical streak through it, especially at low energies. Between a very aggressive clean and the Ga mishap, that mirror has led a hard life. The spare M2 has been recoated, and I plan to install it during 2-bunch time.

The I0 monitor has been replaced with a mini ion chamber of Alastair's design. This chamber works much better than the old paddles and offers the ability to replace about 2" of air with He when doing low energies. Normalization is significantly better than it used to be.

### **Beam motion**

As stated in the last Newsletter, the storage-ring beam was moved. Fortunately, we didn't have to move M1; there was enough adjustment range to bring it in.

We had a problem for a while in which the beam would move up and down every few seconds, even when the EPU wasn't switching. After much complaining to various accelerator people, the problem was traced to some BPMs. These were taken out of the feedback routine and the beam got quiet. However, there are still slow drifts, especially at the top of a fill. When the ring is refilled, it's a good idea to tune M1 before and after the first scan. We also see the effects of switching the EPU polarization. This usually normalizes out, but occasionally doesn't. Tests will be conducted to see if the EPUs change the beam position or only the beam size. The latter is the expected perturbation and nothing can be done about it. The BL 10.0 undulator also causes non-normalizing effects when its gap changes.

## Software

The beamline website is coming together as a useful resource. Bob has put in much time doing this and should be acknowledged for his work.

There has been more work on software. Bob now makes executables of the analysis programs available on the website. The programs now use a new mechanism for the selection of the starting directory for file I/O. They remember where they last got the files, regardless of operations which may have occurred later. Further, the EXAFS programs have a separate memory of file paths from the XY mapping programs.

The XY reader now includes a feature I call masking or population selection. On the scatterplot of one element vs. another, you can select a polygon and then look only at those pixels whose compositions fall in this polygon. Thus, if there are some parts of the sample with a high Cu/Zn ratio and some where the ratio is low, you can select one or the other and see where the high Cu/Zn material is in the sample.

There are auxiliary programs for the XY mapping which allow difference maps, ratio maps, and the ability to take an SCA from one map and insert it into another. Thus, if you want to do chemical-state mapping for Fe, you can do a map at each of two energies near the edge, then insert the Fe map from the high-energy file into the low-energy file, thus giving you a map in which both energies can be seen together.

The non-linear least-squares fit program for EXAFS filtered data is now working. It uses amplitude and phase files from the FT program, or you can provide your own from FEFF (which we don't have on that computer).

The data-taking software has had some changes as well. The biggest thing is that the programs communicate so that they won't let you do two incompatible things at the

same time. The three activities which are mutually incompatible are EXAFS scanning, XY mapping and MCA acquisition. You can run the EXAFS and XY programs, but you can't start a scan if one of the other things is going on.

The old gate-switch error should be a thing of the past. The gate switch is now automatic, so you don't have to remember to throw a physical switch when going between EXAFS and XY scanning.

I'm still way behind in manuals. I have updated some of them to reflect the new realities. I have also written a general one on the dataflow through all the programs.

### **Diffraction**

Diffraction is still not up. We have obtained some hefty stages and the appropriate motion controller (from e-Bay!). What we need is time to set them up in the hutch, string cables, and solve mechanical problems. Real Soon Now!

There is an important difference between 10.3.2 and 7.3.3. Since 7.3.3 was designed from the start as a diffraction line, it has a stage which always moves in the plane of the sample, however the sample is inclined. The 10.3.2 stage was intended for wide-range scanning in EXAFS, with a fixed sample orientation. Thus, if the sample is oriented other than in 'EXAFS orientation', it will move in and out of focus as it scans. Since the optics box takes up most of the space upstream of the sample, diffraction can only be done in reflection mode if the sample is tipped, as on 7.3.3. Thus, diffraction other than in transmission mode carries with it the complication of the loss of focus at the ends of the scan. To fix this would involve a complete redesign of the sample area and the need for new mechanics in which the translation stage is carried on a cradle.

### **Detector**

The 7-element detector seems to have gotten a little gassy, which may be the cause of an elevated background and somewhat poor resolution I've noticed on some elements. We've bought the appropriate fitting to let us pump it out, but we need a three-day no-beam period in which to warm it up, pump it out, then cool it down again.

The new I0 chamber displaces enough air path to make a noticeable difference in the detection of light elements (down to P). We've even detected Si, but that was in quartz grains, so it's not much of a feat. We've borrowed an Amp-Tek detector from 7.3.3 which should do well for EXAFS. Unfortunately, the slow pulses from this detector don't seem to work well with the XIA hardware, so mapping is out. You have to use an old-fashioned SCA. We also intend to try borrowing 7.3.3's 1-element Si detector, which does work well with the XIA hardware.

## **Users**

We have had a string of users since the April shutdown, all of whom have expressed satisfaction with the data quality. These include Scheinost, Scheidegger, and Manceau and the various students and collaborators thereof. We are finally making the transition from the building and upgrade phase to a strong user program. The beamline is booked up pretty solidly through December.